Standard Operating Procedure for the Extraction and Analysis of Semi -Volatile Organic Compounds by Gas Chromatography - Mass Spectrometry

Method Sections

1.0 Scope And Application

1.1 This procedure describes the extraction, analysis, and quantitation of semivolatile organic compounds (SVOCs) in water and soil. The extraction of water samples is based on Method SW846 3510C (Revision 3, December 1996). The extraction of soil samples is based on Method SW846 3550B (Revision 2, December 1996). The analysis of the extracts from both water and soil is base on Method SW846 8270C (Revision 3, December 1996). Internal standards, surrogates, and target compounds that can be determined by this procedure are listed below alphabetically with their Chemical Abstract Service (CAS) number, if available, and the Chemistry Division's LIMS analyte number, if available.

Internal Standards	CAS Number	Analyte Number
Acenaphthene-d_{10}		
Chyrsene-d_{12}		
1,4-Dichlorobenzene-d_4		
Naphthalene-d_8		
Perylene-d_{12}		
Phenanthrene-d_{10}		

Acid Surrogates	CAS Number	Analyte Number
2-Chlorophenol-d_4		
2-Fluorophenol		
Phenol-d_5		42005
2,4,6-Tribromophenol		42255

Base-Neutral Surrogates	CAS Number	Analyte Number
1,2-Dichlorobenzene-d_4		
Nitrobenzene-d_5		42075
2-Fluorobiphenyl		42165
Terpheny-d_{14}		42305

Target Compounds	CAS Number	Analyte Number
Acenaphthene	82-32-9	42190
Acenaphthylene	208-96-8	42180
Aniline	62-53-3	42010
Anthracene	120-12-7	42285
Azobenzene	103-33-3	42250
Benzidine	92-87-5	42370

Target Compounds	CAS Number	Analyte Number

Target Compounds	CAS Number	Analyte Number
Benzoic acid	65-85-0	
Benzo(a)anthracene	56-55-3	42320
Benzo(b)fluoranthene	205-99-2	42340
Benzo(k)fluoranthene	207-08-9	42345
Benzo(g,h,i)perylene	191-24-2	42365
Benzo(a)pyrene	50-32-8	42350
Benzyl alcohol	100-51-6	42040
Bis(2-chloroethoxy)methane	111-91-1	42105
Bis(2-chloroethyl)ether	111-44-4	42020
Bis(2-chloroisopropyl)ether	108-60-1	42055
Bis(2-ethylhexyl)phthalate	117-81-7	42325
4-Bromophenyl phenyl ether	101-55-3	42260
Butyl benzyl phthalate	85-68-7	42310
Carbazole		
4-Chloroaniline	106-47-8	42125
4-Chloro-3-methylphenol	59-50-7	42135
2-Chloronaphthalene	91-58-7	42160
2-Chlorophenol	95-57-8	42025
4-Chlorophenyl-phenyl-ether	7005-72-3	42225
Chrysene	218-01-9	42330
Dibenz(a,h)anthracene	53-70-3	42360
Dibenzofuran	132-64-9	42205
Di-butyl phthalate	84-74-2	42290
1,2-Dichlorobenzene	95-50-1	42045
1,3-Dichlorobenzene	541-73-1	42030
1,4-Dichlorobenzene	106-46-7	42035
3,3'-Dichlorobenzidine	91-94-1	42315
2,4-Dichlorophenol	120-83-2	42110
Diethyl phthalate	84-66-2	42220
2,4-Dimethylphenol	105-67-9	42095
Dimethylphthalate	131-11-3	42175
4,6-Dinitro-2-methylphenol	534-52-1	42240
2,4-Dinitrophenol	51-28-5	42195
2,4-Dinitrotoluene	121-14-2	42210
2,6-Dinitrotoluene	606-20-2	42215
Di - n - octyl phthalate	117-84-0	42335
Fluoranthene	206-44-0	42295
Fluorene	86-73-7	42230

Hexachlorobenzene	118-74-1	42265
Hexachlorobutadiene	87-68-3	42130
Hexachlorocyclopentadiene	77-47-4	42145
Hexachloroethane	67-72-1	42070
Indeno(1,2,3-cd)pyrene	193-39-5	42355
Isophorone	78-59-1	42085
2-Methylnaphthalene	91-57-6	42140
2-Methylphenol	95-48-7	42050
4-Methylphenol	106-44-5	42060
Naphthalene	91-20-3	42120
2-Nitroaniline	88-74-4	42170
3-Nitroaniline	99-09-2	42185
4-Nitroaniline	100-01-6	42235
Nitrobenzene	98-95-3	42080
2-Nitrophenol	88-75-5	42090
4-Nitrophenol	100-02-7	42200
N - Nitrosodimethylamine	62-75-9	
N - Nitrosodiphenylamine	86-30-6	
N - Nitrosodi - n - propylamine	621-64-7	42065
Pentachlorophenol	87-86-5	42270
Phenanthrene	85-01-8	42280
Phenol	108-95-2	42010
Pyrene	129-00-0	42300
1,2,4-Trichlorobenzene	120-82-1	42115
2,4,5-Trichlorophenol	95-95-4	42155
2,4,6-Trichlorophenol	88-06-2	42150

1.2 Reporting levels for analytes determined using this procedure in water are 10 ug/L and 1 ug/g in soils. Analyte detection limits vary by compound. Some are detectable much lower than the reporting level while others are borderline detectable at the reporting level.

2.0 **Summary of Method**

2.1 Water and/or soil samples are prepared via liquid/liquid extraction or ultrasonic extraction. Extract volume is reduced to concentrate analytes and solvent is exchanged, if necessary.

2.2 Samples are injected onto a gas chromatograph(GC)with a mass selective detector(MS). The GC/MS must meet several performance criteria before injecting calibrants and samples. Sample concentrations are determined using the software package with the GC/MS.

3.0 **Definitions**

3.1 Internal standard(IS) - A compound added to the sample extract and calibrants. The IS is used to measure the relative responses of the target compounds and surrogates. The IS is added post extraction and serves to correct any irregularities in the injection volumes.

3.2 Surrogate analyte(Su) - A compound added to the calibrants in known amounts and to the samples prior to extraction. The Su serves to monitor extraction efficiency for individual extractions.

3.3 Target compound - An analyte that can be determined by this procedure.

3.4 Reagent water - Laboratory purified water that is used to prepare all standard, quality control, and blank solutions.

3.5 Laboratory reagent blank(LRB) - an aliquot of reagent water that is extracted and analyzed in the same manner as a water sample. The LRB is used to determine if analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

3.6 Soil blank - A portion of soil suspected to be free of target compounds, internal standards, and surrogates that is extracted in the same manner as a soil sample. The soil blank is used to determine if analytes or other interferences are present in the laboratory environment, reagents, or apparatus and to monitor the extraction efficiency of a “clean” sample.

3.7 Laboratory fortified blank(LFB) - Reagent water to which known quantities of analytes are added in the laboratory. The source of analytes must be different than that used to prepare the standards. The LFB is extracted and analyzed in the same manner as

a sample. It's purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.8 Laboratory fortified soil blank(LFSB) - A soil blank to which know quantities of analytes are added in the laboratory. The source of analytes must be different than that used to prepare the standards. The LFSB is extracted and analyzed in the same manner as a sample. It's purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.9 DFTPP - Decafluorotriphenylphosphine, the compound used to hardware-tune the GC/MS. Specific tuning criteria must be met before sample analysis can begin. See Section 14.4.

3.10 GC/MS Tuning solution - A solution containing 4,4'-DDT, pentachlorophenol, and benzidine that is analyzed to verify injection port inertness and column performance. Specific criteria must be met before analysis can begin. See Section 14.5.

3.11 SPCC's - System Performance Check Compounds - Compounds that are injected to ensure that minimum average response factors are met before the calibration curves are used. Specific criteria must be met before the calibration curves are used. See Section 14.7.

3.12 CCC's - Calibration Check Compounds - Compounds that are analyzed in order to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column or liner. Specific calibration check criteria must be met before the system can be considered suitable for analysis. See Section 14.8.

3.13 Response factor - RF $RF = \frac{\text{Analyte Standard ug/L}}{\text{Analyte Standard Area/IS Area}}$

$$\text{Sample concentration} = RF(ug/L) \times \frac{\text{Analyte Sample Area}}{\text{IS Area}}$$

3.14 Method detection limit(MDL) - The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

4.0 **Interferences**

4.1 Raw analytical data (ion chromatograms) must be evaluated for interferences.

4.2 Contamination by carryover can occur whenever high concentration samples are analyzed. Most samples are of unknown levels, so it must be assumed that they are high concentration samples. Solvent blanks are analyzed after every sample injection to minimize the chance of carryover. Solvent blanks are not required between standard injections.

5.0 Safety

5.1 The toxicity or carcinogenicity of each target compound or reagent has not been precisely determined. Therefore, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 Supplies, Equipment, and Instrumentation

Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other that those specified, but equivalent performance must be demonstrated by the laboratory.

6.1 Supplies and Equipment - Supplies and equipment used in the procedure are common to laboratories and will not be detailed here.

6.2 Instrumentation - Instrumentation in use in this laboratory as of this revision is detailed as follows:

6.2.1 Gas chromatograph (GC): Agilent 6890 Series equipped with ultra high purity helium carrier gas.

6.2.2 GC column: DB-5MS, 30 meters x 0.25mm ID, 0.25u film thickness

6.2.3 Autosampler: Agilent 7683 Series injector

6.2.4 Detector: Agilent 5973 Mass Selective Detector (MSD)

6.2.5 Data acquisition and analysis: Agilent Chemstation Analysis Software

Detailed GC parameters can be found in Section 14.

7.0 **Standards**

This method requires several different standards and check solutions. Their source and purpose will be detailed here. Standard preparation details can be found in Section 12. Quality control solution preparation can be found in Section 13. Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using standard materials other that those specified, but equivalent performance must be demonstrated.

Use/Title	Supelco Cat. Number	Comments
DFTPP	48724 -U	Decafluorotriphenylphosphine at 25000 ug/mL. Diluted to 50 ug/mL and used to hardware tune the GC/MS. See Section 14.4 for performance criteria.
Tuning Solution	47415	Three analytes at 50.0 ug/mL. Injected to assess column performance and injection port inertness. See Section 14.5 for performance criteria.
Acid Surrogates	47261	Four surrogate analytes at 10000 ug/mL each
Base/Neutral Surrogates	47262	Four surrogate analytes at 5000 ug/mL each
Acids Spike Mix	48878	Five analytes at 2000 ug/mL each for matrix spikes.
Base/Neutral Spike Mix	48100-U	Six analytes at 1000 ug/mL each for matrix spikes.

Use/Title	Supelco Cat. Number	Comments
Internal Standards	47906	Six internal standards at 4000 ug/mL each.
Calibration Mix	506508	Contains 64 of the 70 target analytes at 1000 ug/mL each listed in Section 1.1. Does not contain aniline, benzidine, benzoic acid, benzyl alcohol, 3,3' - dichlororbenzidine, and N - nitrosodiphenylamine.
Hazardous Substances Mix 1	48907	Contains 4 analytes, including benzoic acid at 2000 ug/mL each.
Hazardous Substances Mix 2	48908	Contains 8 analytes, including aniline and benzyl alcohol, at 2000 ug/mL each.
Benzidines Mix	48906	Contains benzidine and 3,3' - dichloro-benzidine at 2000 ug/mL each
N - Nitrosodi-phenylamine	40060	Contains N - Nitrosodiphenylamine at 5000 ug/mL.
Acid Calibration Check Compounds	47386	Six analytes at 2000 ug/mL each for acid calibration check. See Section 14.8 for performance criteria.
Base/Neutrals Calibration Check Compounds	48464	Seven analytes at 2000 ug/mL each for base/neutrals calibration check. See for performance criteria.
System Performance Check Compounds	47390-U	Four analytes at 1000 ug/mL each for system performance check. See Section 14.7 for performance criteria.

8.0 Sample Collection, Preservation, Shipping, and Storage

8.1 The sample collection procedure depends upon the sample matrix. The following table describes the procedures for various sample types.

Sample Matrix	Container	Preservative	Holding Time
Aqueous with no residual chlorine	1 liter amber glass with Teflon-lined lid	Cool to 4 degrees C	7 days to extract 40 days to analyze after extraction.
Aqueous with residual chlorine	1 liter amber glass with Teflon-lined lid	Cool to 4 degrees C Add 3mL 10% sodium thiosulfate per gallon.	7 days to extract 40 days to analyze after extraction.
Solid samples	Widemouth glass with Teflon-lined lid	Cool to 4 degrees C	14 days to extract 40 days to analyze after extraction.
Concentrated Waste Samples	Widemouth glass with Teflon-lined lid	None	14 days to extract 40 days to analyze after extraction.

Source of sample collection information: SW846, Revision 3, December 1996
Chapter 4, Section 4.1.2, page 7

8.2 Sample extracts are stored at -10 degrees C, protected from light, in GC autosampler vials.

9.0 Quality Control

Quality control must be demonstrated by the analyst initially before the analysis of samples and with each sample extraction set.

9.1 Initial Demonstration of Capability - The laboratory must demonstrate initial proficiency with this procedure by generating data of acceptable accuracy and precision for the target analytes in a clean matrix.

9.1.1. Extract and analyze a minimum of 4 LFB's. See Section 13 for preparation of LFB solutions and Section 10 for extraction procedure.

9.1.2 Determine the average recovery of each analyte in each LFB.

9.1.3 Recoveries of analytes should be between 70 and 130%. Due to the large number of analytes and the nature of this extraction procedure, it is likely that several analytes will fail to be within this range. Table 2 in Section 18 lists average recoveries of all analytes in LFB's that have been extracted to date using this procedure. These average recoveries are useful comparisons to individual LFB recoveries to evaluate extraction efficency.

9.2 Extraction set quality control - The analyst must perform several quality control checks with each sample extraction set. Those include but are not limited to:

9.2.1 Extraction and analysis of LRB. If any analyte in the LRB exceeds the reporting limit of 10 ug/L, corrective action must be taken to locate and reduce the source of the contamination.

9.2.2 Extraction and analysis of LFB - See Section 10 for LFB preparation. Analytes recoveries should be within 70 to 130% or within range of the average recoveries found in Table 2 in Section 18.

9.2.3 Analysis of DFTPP to hardware tune the system. Performance criteria can be found in Section 14.4.

9.2.4 Analysis of the tuning solution to establish that system is performing acceptably from the chromatographic standpoint. Performance criteria can be found in Section 14.5.

9.2.5 Analysis of a "tube spike", a solution at the same concentrations as the LFB analytes, but not extracted. LFB aliquots are added and diluted directly to 1mL final solvent. The purpose of this is to verify calibration curve performance and can take the place of the CCC.

9.2.6 The preparation and analysis of SPCCs (system performance check compounds) may be necessary for some samples. See Section 13 for preparation and performance criteria.

9.2.7 The preparation and analysis of CCCs (calibration check compounds) may be necessary for some samples. See Section 13 for preparation and performance criteria.

10.0 **Extraction Procedure for Water Samples**

10.1 Accurately measure and transfer the volume of water to be extracted into a two-liter separatory funnel. For samples expected to have low level analyte concentrations, one liter of water should be extracted. If the samples are expected to have higher levels of analytes, lower volumes should be extracted.

10.2 Adjust the pH of samples to less than 2 using concentrated H_2SO_4 (sulfuric acid). Five mL is typical of the volume needed to adjust the pH to less than 2. The addition of excess acid has been shown to contribute to lower analyte recoveries in the LFB's, so monitor the pH as the acid is added to avoid the addition of excess acid.

10.3 Add 10 uL acid surrogates to all extractions, Supelco Number 4-7261.

Final concentration = 10000 ug/mL x 10uL/1000uLfinal volume x 1mL/1L sample
= 100 ug/L each

10.4 Add 50 uL LFB stock to the LFB extraction and to any spiked samples. LFB Stock = Supelco Number 506508. The stock used for this preparation must be different than the stock used to prepare the standards. This stock is typically used instead of the acid calibration checks and the base/neutral checks because it has significantly more analytes.

Final LFB/spike concentrations = 1000 ug/mL x 50uL/1000uL x 1mL/1L sample
= 50 ug/L each

10.5 Add 60 mL methylene chloride and shake the sample for 2 minutes. Allow the phases to separate.

10.6 Drain the lower methylene chloride phase into flask for solvent evaporation.

10.7 Repeat steps 10.5 and 10.6 two more times using the same flask to collect the solvent.

10.8 Reduce the volume of methylene chloride in the flask.

10.9 Adjust the pH of the water in separatory funnel to greater than 11 using concentrated NaOH (sodium hydroxide). Fifty mL is typical of the volume NaOH needed for the adjustment. The addition of excess base has been shown to contribute to lower analyte recoveries in the LFB's, so monitor the pH as the base is added to avoid the addition of excess base.

10.10 Add 10 uL base/neutral surrogates to all extractions, Supelco Number 4-7262.

Final concentration = 5000 ug/mL x 10uL/1000uL final volume x 1mL/1L sample
= 50.0 ug/L each

10.11 Add 60 mL methylene chloride and shake the sample for 2 minutes. Allow the phases to separate.

10.12 Drain the methylene chloride phase into the same flask that was used in step 10.6.

10.13 Repeat steps 10.11 and 10.12 two more times using the same flask to collect the methylene chloride so all of the extract of a sample is in a single flask.

10.14 Pass the extract over sodium sulfate in a funnel to remove excess water if necessary.

10.15 Reduce the volume of methylene chloride to less than 1 mL in the flask.

10.16 Add 10 uL internal standards solution, Supelco Number 4-7906.
Final concentrations = 4000 ug/mL x 10uL/1000uL = 40 ug/mL

10.17 Adust final volume of solvent to 1mL. Transfer the sample to a GC vial for analysis.

11.0 **Extraction Procedure for Solid Samples**

11.1 Accurately weigh the sample into a glass or Teflon 400 mL beaker. If low level detections are expected, weigh 30 grams sample. If higher levels are expected, lower sample weights should be used.

11.2 If necessary, add enough sodium sulfate to form a “free flowing” powder.

11.3 Add 10 uL acid surrogates to all extractions, Supelco Number 4-7261.
Final concentration is dependent upon sample weight. Ten grams will be used here for illustration. Final concentration =

10000 ug/mL x 0.01mL/10 g = 10.0 ug/g each.

11.4 Add 10 uL base/neutral surrogates to all extractions, Supelco Number 4-7262.

Final concentration =

5000 ug/mL x 0.01mL/10 g = 10.0 ug/g each.

11.5 Add 50 uL LFB stock to the LFB extraction and to any spiked samples. LFB Stock = Supelco Number 506508. The stock used for this preparation must be different than the stock used to prepare the standards. This stock is typically used instead of the acid calibration checks and the base/neutral checks because it has significantly more analytes. Final concentration =

1000 ug/mL x 0.05mL/10g = 5 ug/g each.

11.6 Extract by sonication for 3 minutes with 100 mL acetone/hexane 1/1.

11.7 Filter the extract while transferring to a suitable flask for solvent reduction.

11.8 Repeat steps 11.6 and 11.7 two more times, combining the extracts for a sample in the same flask for solvent reduction.

11.9 Reduce the volume of methylene chloride to less than 1 mL in the flask.

11.10 Add 10 uL internal standards solution, Supelco Number 4-7906. Final concentration = 4000 ug/mL x 0.01 mL/10 g = 4 ug/g.

11.11 Adjust final volume of solvent to 1mL. Transfer the sample to a GC vial for analysis.

12.0 **Standard Preparation**

Standard preparation involves the use of several stock solutions since no single stock contains all of the target compounds. Two separate sets of calibrants are prepared.

12.1 Preparation of 506508 standards - Supelco stock solution 506508 contains 64 of the 70 target compounds listed in Section 1. The stock solution used to prepare the standards must not be the same as the stock used to prepare the LFB's and spikes. Preparation in methylene chloride is as follows:

Std	Dilution	Concentration
1	10.0uL/1.0mL	1000 ug/mL x 0.01mL/1.0mL = 10.0 ug/mL
2	25.0uL/1.0mL	1000 ug/mL x 0.025mL/1.0mL = 25.0 ug/mL
3	50.0uL/1.0mL	1000 ug/mL x 0.050mL/1.0mL = 50.0 ug/mL
4	75.0uL/1.0mL	1000 ug/mL x 0.075mL/1.0mL = 75.0 ug/mL
5	100.0uL/1.0mL	1000 ug/mL x 0.100mL/1.0mL = 100.0 ug/mL

To all standard solutions, add the following:

1. 10.0 uL acid surrogates, Supelco 4-7261, final concentration = 10000 ug/mL x 10uL/1000uL = 100 ug/mL

2. 10.0 uL base/neutral surrogates, Supelco 4-7262, final concentration = 5000 ug/mL x 10uL/1000uL = 50 ug/mL

3. 10.0 uL internal standards, Supleco 4-7906, final concentration = 4000 ug/mL x 10uL/1000uL = 40.0 ug/mL

Dilute to 1mL and transfer the solutions to GC vials for analysis.

12.2 Preparation of "Haz Mix" standard - The 6 target compounds not found in Std 506508 are found in 4 different stock solutions, two of which are called Hazardous Mix,

hence the name "Haz Mix". These 4 stocks may contain other target compounds, but since they are also in Std 506508, they need not be included for calibration purposes. The preparation of a complete calibration curve for these analytes takes significant preparation time for just the 6 analytes. A single calibrant is prepared at 50 ug/mL to determine sample concentrations, if any for these target compounds. The standard name and target compounds they contain are as follows:

Standard	Supelco No.	Target Compounds	Concentration
Hazardous Substances Mix 1	4-8907	benzoic acid	2000 ug/mL
Hazardous Substances Mix 2	4-8908	aniline benzyl alcohol	2000 ug/mL each
Benzidines Mix	4-8906	benzidine 3,3'-dichlorobenzidine	2000 ug/mL each
N - Nitroso-diphenylamine	4-0060	N - Nitrosodiphenylamine	5000 ug/mL

Preparation of the Haz Mix standard in methylene chloride is as follows:

In a single 1 mL flask add, 25.0 uL each of Hazardous Substances Mix1, Hazardous Substances Mix 2, and the benzidines mix. Into the same flask, add 10.0 uL of the N - Nitrosodiphenyamine stock. The concentration of each analyte is:

2000 ug/mL x 25.0uL/1000uL = 50.0 ug/L

or

5000 ug/mL x 10.0uL/1000uL = 50.0 ug/L

Then add to the 1 mL flask,

1. 10.0 uL acid surrogates, Supelco 4-7261, final concentration = 10000 ug/mL x 10uL/1000uL = 100 ug/mL

2. 10.0 uL base/neutral surrogates, Supelco 4-7262, final concentration = 5000 ug/mL x 10uL/1000uL = 50 ug/mL

3. 10.0 uL internal standards, Supleco 4-7906, final concentration = 4000 ug/mL x 10uL/1000uL = 40.0 ug/mL

Dilute to 1mL and transfer the solution to a GC vial for analysis.

13.0 **Quality Control Solution Preparation**

Several quality control solutions are required for this analysis. Performance criteria can be found in Section 14, Data Acquisition.

13.1 DFTPP preparation - DFTPP is used to hardware tune the GC/MS. The DFTPP stock solution, Supelco Number 48724-U, is at a concentration of 25000 ug/mL and is prepared in methylene chloride as follows:

Dilute 2uL/1mL; Final concentration = 25000 ug/mL x 2uL/1000uL = 50 ug/mL.

Transfer the solution to a GC vial for analysis.

Performance criteria can be found in Section 14.4.

13.2 GC/MS tuning solution preparation - The GC/MS tuning solution compounds, Supelco Number 47415, are at 50.0 ug/mL and need no dilution. Transfer the contents of a stock ampule to a GC vial for analysis. Performance criteria can be found in Section 14.5.

13.3 LFB preparation - Add 50uL LFB Stock to 1L water prior to extraction and extract as any other sample. LFB Stock = Supelco Number 506508. The stock used for this preparation must be different than the stock used to prepare the standards. This stock is typically used instead of the acid calibration checks and the base/neutral checks because it has significantly more analytes.

Final LFB/spike concentrations = 1000 ug/mL x 50uL/1000uL x 1mL/1L sample
= 50 ug/L each

Measured concentrations of the analytes should be within 70 to 130% of 50.0 ug/L.

Historical LFB recoveries are listed in Section 18, Table 2.

13.4 SPCC preparation - The SPCC solution contains 4 target compounds at 1000 ug/mL each that must meet a minimum response factor or 0.050. The SPCC stock, Supelco Number 47390-U, can be diluted in methylene chloride to various concentrations

as detailed below:

SPCC	Dilution	Concentration
1	10.0uL/1.0mL	1000 ug/mL x 0.01mL/1.0mL = 10.0 ug/mL
2	25.0uL/1.0mL	1000 ug/mL x 0.025mL/1.0mL = 25.0 ug/mL
3	50.0uL/1.0mL	1000 ug/mL x 0.050mL/1.0mL = 50.0 ug/mL
4	75.0uL/1.0mL	1000 ug/mL x 0.075mL/1.0mL = 75.0 ug/mL
5	100.0uL/1.0mL	1000 ug/mL x 0.100mL/1.0mL = 100.0 ug/mL

To all SPCC solutions, add the following:

1. 10.0 uL acid surrogates, Supelco 4-7261, final concentration = 10000 ug/mL x 10uL/1000uL = 100 ug/mL

2. 10.0 uL base/neutral surrogates, Supelco 4-7262, final concentration = 5000 ug/mL x 10uL/1000uL = 50 ug/mL

3. 10.0 uL internal standards, Supleco 4-7906, final concentration = 4000 ug/mL x 10uL/1000uL = 40.0 ug/mL

Dilute to 1mL and transfer to GC vials for analysis.

Performance criteria can be found in Section 14.7.

13.5 “Tube spike” preparation - A “tube spike” is prepared and analyzed to evaluate the calibration curves. This can be done instead of a CCC evaluation. “Tube Spike” preparation in methylene chloride is as follows: Into a single graduated centrifuge tube add 50uL LFB stock, Supelco Number 506508. Final concentration =

1000 ug/mL x 50uL/1000uL = 50 ug/L each target compound.

Then add to the same tube,

1. 10.0 uL acid surrogates, Supelco 4-7261, final concentration = 10000 ug/mL x 10uL/1000uL = 100 ug/mL

2. 10.0 uL base/neutral surrogates, Supelco 4-7262, final concentration = 5000 ug/mL x 10uL/1000uL = 50 ug/mL

3. 10.0 uL internal standards, Supleco 4-7906, final concentration = 4000 ug/mL x 10uL/1000uL = 40.0 ug/mL

Dilute to 1 mL and transfer to a GC vial for analysis.

Performance criteria can be found in Section 14.9. Historical "tube spike" recoveries are listed in Section 18, Table 3.

13.6 CCC solution preparation - Calibration check compound solutions can be analyzed instead of the "tube spike" This laboratory will normally analyze a "tube spike"instead of the CCCs because more target compounds are evaluated using the tube spike. The CCC's are subject to more rigorous performance criteria, but adequate LFB recoveries are acceptable in every other method used in this laboratory and will be considered so for this procedure also. CCC preparation will be included here in the event the analyst desires to use them instead of the "tube spike". The procedure is as follows:

13.6.1 Prepare the calibration check standards at five different concentrations in 1mL volumetric flasks in methylene chloride as shown below.

Stock solutions: Supelco 47386, Acid Cal Check Compounds
Supelco 48464, Base/Neutral Check Compounds

CCC	47386uL	48464uL	Final concentration
1	5.0uL	5.0uL	10.0 ug/L each
2	12.5uL	12.5uL	25.0 ug/L each
3	25.0uL	25.0uL	50.0 ug/L each
4	37.5uL	37.5uL	75.0 ug/L each
5	50.0uL	50.0uL	100.0 ug/L each

Then add to the each tube,

1. 10.0 uL acid surrogates, Supelco 4-7261, final concentration = 10000 ug/mL x 10uL/1000uL = 100 ug/mL

2. 10.0 uL base/neutral surrogates, Supelco 4-7262, final concentration = 5000 ug/mL x 10uL/1000uL = 50 ug/mL

3. 10.0 uL internal standards, Supleco 4-7906, final concentration = 4000 ug/mL x 10uL/1000uL = 40.0 ug/mL

Dilute to 1mL and transfer the solutions to GC vials for analysis.

Performance criteria can be found in Section 14.8.

14.0 **Data Acquisition, Analysis, and QC Performance Criteria**

14.1 Data acquisition and analysis is a multi-step process. As of this revision this laboratory uses and Agilent 6890 Series GC, an Agilent 7683 Series autosampler, an Agilent 5973 MSD, and Agilent Chemstation Analysis Software. Method conditions will be listed here.

GC column: DB5-MS, 30 meters x 0.25 mm, 0.25u film thickness
Temperature program: 40 degrees C for 1 min
15 degrees C/min to 120 degrees C, hold for 1 minute
10 degrees C/min to 270 degrees C, hold for 0 minutes
30 degrees C/min to 300 degrees C, hold for 7 minutes
Total run time = 30.33 minutes

Mode: Constant flow, 1.1 mL/min, splitless
PSI at initial conditions: about 8.1
Liner: Single Gooseneck Silcosleeve with Carbofrit; Restek Number 11813-702-949
Injection port temp: 220 degrees C
Injection volume: 1 uL
Purge flow: 50.0 mL/min
Purge time: 0.50 min
Total flow: 53.9 mL/min
Transfer line: Auxiliary 2; Temperature: 280 degrees C
Solvent delay: 3.00 min
Scan Range: 45 - 450 amu

14.2 Familiarity with the Agilent Chemstation Software will be assumed.

14.3 Create a method by copying the most recent method to the current date using the format: SVyymmdd for example - SV030415. Method name length is limited to 8 characters. Autotune the system using DFTPP autotune. Save tune file printout for documentation.

14.4 After successfully tuning the instrument, create an analysis sequence and analyze the DFTPP solution at 50.0 ug/mL prepared in Section 13.1. The DFTPP solution should be analyzed at the beginning and end of each analysis to ensure the adequate performance over the course of the sequence. If the analysis is very long, the DFTPP should be injected at regular intervals throughout the analysis. Performance criteria are:

Target Mass	Relative to Mass	Lower Limit %	Upper Limit%
51	198	30	60
68	69	0.00	2
69	198	0.00	100
70	69	0.00	2
127	198	40	60
197	198	0.00	1
198	198	100	100
199	198	5	9
275	198	10	30
365	198	1	100
441	443	0.01	100
442	198	40	100
443	442	17	23

A chromatogram of a DFTPP solution is shown is Section 18, Chromatograms.

14.5 Once the DFTPP has been successfully analyzed, inject and analyze the GC/MS tuning solution to assess GC column performance and injection port inertness. Tuning solution preparation is described in Section 13.2 Performance criteria for the tuning solution are:

14.5.1 Degradation of DDT to DDE and DDD should not exceed 20%

14.5.2 Benzidine and pentachlorophenol should be present at their normal responses and peak tailing should be minimal.

A chromatogram of a tuning solution injection is shown in Section 18, Chromatograms.

14.6 Inject all standard solutions and use them to construct calibration curves for the target compounds.

A chromatogram of an injection of standard solution of Supelco 506508 is shown in Section 18, Chromatograms.

14.7 Inject and analyze the SPCC solutions prepared in Section 13.4 to ensure that the minimum average response factors are met before the calibration curves are used. The 4 SPCCs have very low response factors and tend to decrease as the chromatographic system or the standard material begins to deteriorate. They are typically the first to show poor performance and the must meet the minimum requirements when the system is calibrated. Performance criteria for the SPCC compounds are that the average response factor must be at least 0.050.

A chromatogram of SPCC's is shown in Section 18, Chromatograms.

14.8 Inject and analyze(if necessary) the CCC solutions prepared in Section 13.6. Calculate the average response factor and the relative standard deviation(RSD) of the response factors for each of the target compounds in the CCCs. Performance criteria is that the RSD's of the target compounds must be less than 30%.

A chromatogram of the CCC's is shown in Section 18, Chromatograms.

14.9 Inject and analyze the "tube spike" prepared in Section 13.5, if used in the analysis. The purpose of the "tube spike" is to evaluate the calibration curves integrity. Generally, percent recovery of the "tube spike" compounds should be between 70 and 130%. Due to the large number of compounds, some recoveries outside the limits can be expected. The analyst should document recoveries and look for trends over the course of several analyses. Re-inject the "tube spike"at regular intervals over the course of the analysis. Historical "tube spike"recoveries as listed in Section 18, Table 3.

14.10 After successful analysis of the standards and QC solutions, analyze the sample extracts and the QC extracts(LRB, LFB, matrix spike, matrix duplicate). Re-inject the standards at the end of the analysis. If all QC is acceptable with the first set of standards the second set does not need to be included in the curves.

15.0 Method Performance

15.1 The reporting level for all target compounds is 10.0 ug/L, assuming that 1 liter of sample is extracted. This is equivalent to the lowest standard solution to be analyzed. This level must be analyzed and shown to be detectable before 10.0 ug/L can be used as the reporting level.

15.2 The analyst should document "tube spike" and LFB recoveries and be alert for trends over the course of analyses. While some recoveries may be outside the 70 to 130% limits, this may be normal and the results can be considered valid.

15.3 The analyst should use caution when adjusting pH. The addition of excess acid or base has been shown to have detrimental effects on target compound recoveries so the pH should be monitored so the pH requirements are not exceeded.

16.0 Pollution Prevention

16.1 This method uses significant amounts of organic solvents. Waste should be minimized by recycling solvents if possible. Recovered solvents should be recycled or properly disposed of.

17.0 Waste Management

17.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions. The laboratory has the responsibility to protect the environment by minimizing and controlling all releases form fume hoods and bench operations.

18.0 Tables and Chromatograms

18.1 Table 1: Analyte retention times, quant ions, and qual ions
18.2 Table 2: Historical LFB recoveries
18.3 Table 3: Historical "tube spike" recoveries
18.4 Chromatograms
- 18.4.1 Standard 506508
- 18.4.2 Internal standards
- 18.4.3 Acid surrogates
- 18.4.4 Base/Neutral Surrogates
- 18.4.5 DFTPP
- 18.4.6 Tune solution
- 18.4.7 LRB

Table 1 Retention Times and Primary and Secondary Ions for Internal Standards, Surrogates, and Target Compounds

Compound type: IS = Internal standard
AS = Acid surrogate
BNS = Base/Neutral Surrogate

All others are target compounds

Target compounds listed in order of elution, although some shifts occur with different columns and phases. The internal standard that precedes a surrogate or target compound is applied to that surrogate or target. For example, Peak 1 is the internal standard that applies to peak 13 and Peak 20 is the internal standard that applies to peak 25.

Peak	Compound(type)	R_t(min)	Quant Ion	Qualifier Ions (Percent Q Ion)
1	1,4 - dichlorobenzene -d_4 (IS)	6.30	152.0	115(72.4)
2	2-fluorophenol (AS)	4.65	112.1	64.1(50.8), 92.0(23.8)
3	phenol - d_5 (AS)	5.79	99.1	71.1(28.3)
4	2-chlorophenol - d_4 (AS)	6.00	132.0	134.0(32.8), 96.1(12.8)
5	1,2-dichlorobennzene - d4 (BNS)	6.53	150.0	152.0(63.4), 115.0(38.7)
6	N - Nitrosodimethylamine	3.31	74.0	42.0(68.0), 75.0(2.5)
7	Aniline	5.83	93.1	66.1(34.6), 65.1(17.2)
8	Phenol	5.81	94.0	66.1(29.6), 65.1(23.9)
9	Bis(2-chloroethyl) ether	5.95	93.0	63.0(65.3), 95.0(32.6)
10	2-Chlorophenol	6.02	128.0	64.0(38.4), 130.0(32.8)
11	1,3-Dichlorobenzene	6.23	146.0	148.0(63.6), 111.0(38.5)
12	1,4-Dichlorobenzene	6.34	146.0	147.9(64.4), 111.0(36.3)
13	1,2-Dichlorobenzene	6.55	146.0	147.9(64.0), 111.0(39.4)
14	Benzyl alcohol	6.49	79.1	108.1(90.1), 107.0(62.8)
15	2-Methylphenol	6.67	108.1	107.1(83.7), 79.1(33.1)
16	Bis(2-chloroisopropyl) ether	6.72	121.0	77.0(47.7), 79.0(36.6)
17	4-Methylphenol	6.92	107.1	108.1(85.1), 77.1(26.0)
18	Hexachloroethane	7.10	200.8	116.9(107.3), 202.8(63.7)
19	N - Nitroso-di - n - propyl amine	6.94	130.1	70.1(310.1), 71.1(54.7)
20	Naphthalene - d_8 (IS)	8.62	136.1	108.1(10.1)
21	Nitrobenzene - d_5 (BNS)	7.18	128.1	82.1(186.1), 54.1(78.8)
22	Nitrobenzene	7.22	123.0	77.1(176.2), 51.1(74.5)
23	Isophorone	7.69	82.1	138.1(24.1), 54.1(11.1)
24	2-Nitrophenol	7.84	139.0	65.1(35.3), 81.0(25.0)

Peak	Compound(type)	R_t(min)	Quant Ion	Qualifier Ions (Percent Q Ion)
25	2,4-Dimethylphenol	7.94	122.1	107.1(101.0), 121.1(54.7)
26	Bis(2-chloroethoxy) methane	8.16	93.0	63.0(58.1), 95.0(32.8)
27	Benzoic acid	8.13	105.0	122.0(87.5), 77.0(69.8)
28	2,4-Dichlorophenol	8.32	162.0	164.0(63.1), 98.0(32.4)
29	1,2,4-Trichlorobenzene	8.51	179.9	181.9(94.0), 145.0(29.6)
30	Naphthalene	8.67	128.1	127.1(14.2), 102.1(9.3)
31	4-Chloroaniline	8.81	127.0	129.0(32.7), 65.1(23.3)
32	Hexachlorobutadiene	8.97	224.8	226.8(62.8), 222.8(63.6)
33	4-Chloro-3-methylphenol	9.94	107.1	142.0(91.5), 77.1(45.3)
34	2-Methylnaphthalene	10.24	142.1	141.1(85.3), 115.1(33.3)
35	Acenaphthene-d_{10} (IS)	12.71	162.2	164.2(106.1), 160.1(48.2)
36	2-Fluorobiphenyl (BNS)	11.15	172.1	171.1(35.8), 170.1(24.5)
37	Hexachlorocyclopentadiene	10.62	236.8	238.8(63.7), 234.8(64.7)
38	2,4,6 - Trichlorophenol	10.92	195.9	197.9(93.6), 97.0(61.8)
39	2,4,5 - Trichlorophenol	10.98	195.9	197.9(96.6), 97.0(38.8)
40	2-Chloronaphthalene	11.38	162.0	127.1(42.1), 164.0(34.1)
41	2-Nitroanaline	11.66	138.0	92.1(53.8), 65.1(69.7)
42	Dimethylphthalate	12.19	163.1	77.1(19.2), 135.0(5.9)
43	Acenaphthylene	12.36	152.1	151.1(21.3), 153.1(14.0)
44	2,6-Dinitrotoluene	12.29	165.0	89.1(48.6), 63.1(42.7)
45	3-Nitroaniline	12.66	138.1	65.1(98.3), 92.1(100.2)
46	Acenaphthene	12.79	153.1	154.1(94.5), 152.1(49.9)
47	2,4-Dinitrophenol	12.92	184.0	154.0(46.4), 63.0(46.9)
48	4-Nitrophenol	13.13	65.1	139.0(123.8), 109.0(72.0)
49	Dibenzofuran	13.21	168.1	139.1(40.4), 169.1(14.2)
50	2,4-Dintrotoluene	13.25	165.0	89.1(61.9), 63.1(33.0)
51	Tetrachlorophenol	13.50	231.8	229.8(76.8), 130.9(46.1)
52	Diethyl phthalate	13.94	149.0	177.1(24.3), 150.0(12.9)
53	Fluorene	14.04	166.1	165.1(92.4), 82.6(13.9)
54	4-Chlorophenyl-phenyl ether	14.12	204.0	206.0(33.5), 141.1(56.1)
55	4-Nitroaniline	14.13	138.0	92.1(41.6), 65.1(83.4)
56	Phenanthrene-d_{10} (IS)	16.29	188.2	94.1(14.1), 160.1(11.1)
57	2,4,6-Tribromophenol (AS)	14.61	329.8	331.8(94.2), 62.1(69.6)
58	N - Nitrosodiphenylaime	14.41	166.0	65.0(132.0), 141.0(97.0)
59	4,6-Dinitro-2-methylphenol	14.21	198.0	105.0(46.8), 121.0(41.4)
60	Azobenzene	14.49	77.1	182.1(37.6), 105.1(21.4)

Peak	Compound(type)	R_t(min)	Quant Ion	Qualifier Ions (Percent Q Ion)
61	4-Bromophenyl-phenyl ether	15.29	248.0	250.0(94.4), 168.1(15.2)
62	Hexachlorobenzene	15.35	283.8	285.8(78.5), 248.9(32.0)
63	Pentachlorophenol	15.86	265.8	263.8(63.9), 267.8(64.4)
64	Phenanthrene	16.34	178.1	89.1(12.0), 177.1(11.5)
65	Anthracene	16.47	178.1	89.1(13.8), 177.1(10.0)
66	Carbazole	16.91	167.1	166.1(21.7), 168.1(14.4)
67	Dibutyl phthalate	17.95	149.1	150.1(10.4), 104.0(5.6)
68	Fluoranthene	19.21	202.1	101.1(18.8), 201.1(15.1)
69	Chrysene-d_{12} (IS)	22.63	240.2	236.2(27.2), 241.2(20.1)
70	Terphenyl-d_{14} (BNS)	20.25	244.3	245.3(20.4), 240.2(10.4)
71	Benzidine	19.64	184.1	185.1(14.8), 183.1(11.2)
72	Pyrene	19.73	202.1	200.1(21.1), 203.1(18.3)
73	Benzyl butyl phthalate	21.54	149.0	91.1(67.9), 206.1(24.3)
74	Benzo(a) anthracene	22.62	228.1	226.1(28.0), 229.1(20.6)
75	3,3'-Dichlorobenzidine	22.65	252.0	254.0(63.5), 182.1(11.3)
76	Chrysene	22.69	228.1	226.1(30.6), 229.1(19.9)
77	Bis(2-ethylhexyl) phthalate	22.94	149.1	167.0(32.9), 71.1(17.7)
78	Di - n - octyl phthalate	24.08	149.0	150.0(10.6), 57.1(7.2)
79	Perylene-d12 (IS)	25.32	264.2	260.1(24.1), 265.2(22.1)
80	Benzo(b)fluoranthene	24.57	252.1	250.1(23.1), 253.1(22.4)
81	Benzo(k)fluoranthene	24.63	252.1	250.1(22.5), 253.1(21.4)
82	Benzo(a)pyrene	25.20	252.1	250.1(24.3), 126.1(23.7)
83	Indeno(1,2,3-cd)pyrene	27.91	276.1	277.1(24.7), 274.1(20.0)
84	Dibenz(a,h)anthracene	28.02	278.1	279.1(23.9), 139.0(22.2)
85	Benzo(g,h,i)perylene	28.68	276.1	138.0(35.5), 274.0(21.4)

Table 2: Historical LFB recoveries

		Mean Recovery	N	Range
1	1,4 - Dichlorobenzene -d_4 (IS)	101%	5	90 -104%
2	2-Fluorophenol (AS)	37	5	7 - 58
3	Phenol - d_5 (AS)	31	5	6 - 58
4	2-Chlorophenol - d_4 (AS)	58	5	12 - 86
5	1,2-Dichlorobennzene - d4 (BNS)	54	5	9 - 84
6	N - Nitrosodimethylamine	51	3	9 - 76
7	Aniline			
8	Phenol	33	5	6 - 60
9	Bis(2-chloroethyl) ether	65	3	12 - 97
10	2-Chlorophenol	59	5	12 - 91
11	1,3-Dichlorobenzene	52	3	7 - 52
12	1,4-Dichlorobenzene	49	5	7 - 83
13	1,2-Dichlorobenzene	54	3	7 - 54
14	Benzyl alcohol			
15	2-Methylphenol	64	3	12 - 91
16	Bis(2-chloroisopropyl) ether	63	3	11 - 94
17	4-Methylphenol	62	3	13 - 89
18	Hexachloroethane	55	3	6 - 93
19	N - Nitroso-di - n - propyl amine	71	4	15 - 101
20	Naphthalene - d_8 (IS)	85	5	9 - 122
21	Nitrobenzene - d_5 (BNS)	65	5	13 - 98
22	Nitrobenzene	65	3	13 - 97
23	Isophorone	73	3	21 - 100
24	2-Nitrophenol	67	3	13 - 101
25	2,4-Dimethylphenol	69	3	16 - 96
26	Bis(2-chloroethoxy) methane	70	3	14 - 101
27	Benzoic acid			
28	2,4-Dichlorophenol	70	3	15 - 104
29	1,2,4-Trichlorobenzene	58	5	8 - 88
30	Naphthalene	61	3	10 - 92
31	4-Chloroaniline	66	3	18 -92
32	Hexachlorobutadiene	56	3	7 - 86
33	4-Chloro-3-methylphenol	77	5	45 - 109
34	2-Methylnaphthalene	65	3	13 - 94

		Mean Recovery	N	Range
35	Acenaphthene-d_{10} (IS)	104	5	91 - 122
36	2-Fluorobiphenyl (BNS)	65	5	20 - 97
37	Hexachlorocyclopentadiene	71	3	14 - 108
38	2,4,6 - Trichlorophenol	80	3	38 - 109
39	2,4,5 - Trichlorophenol	82	3	48 - 105
40	2-Chloronaphthalene	70	3	21 - 96
41	2-Nitroanaline	93	3	66 - 93
42	Dimethylphthalate	95	3	78 - 105
43	Acenaphthylene	76	3	34 - 99
44	2,6-Dinitrotoluene	97	3	78 - 110
45	3-Nitroaniline	89	3	73 - 102
46	Acenaphthene	71	5	37 - 98
47	2,4-Dinitrophenol	51	3	1 - 51
48	4-Nitrophenol	46	5	15 - 75
49	Dibenzofuran	82	3	47 - 101
50	2,4-Dintrotoluene	88	5	55 - 113
51	Tetrachlorophenol			
52	Diethyl phthalate	100	3	93 - 105
53	Fluorene	88	3	62 - 103
54	4-Chlorophenyl-phenyl ether	87	3	60 - 103
55	4-Nitroaniline	99	3	92 - 106
56	Phenanthrene-d_{10} (IS)	105	5	92 - 123
57	2,4,6-Tribromophenol (AS)	84	5	48 - 104
58	N - Nitrosodiphenylaime			
59	4,6-Dinitro-2-methylphenol	78	3	31 - 111
60	Azobenzene	93	3	71 - 107
61	4-Bromophenyl-phenyl ether	94	3	77 - 104
62	Hexachlorobenze	97	3	86 - 104
63	Pentachlorophenol	86	5	58 - 117
64	Phenanthrene	100	3	89 - 108
65	Anthracene	98	3	89 - 104
66	Carbazol	104	3	98 - 109
67	Dibutyl phthalate	102	3	103 - 109
68	Fluoranthene	105	3	99 - 105

		Mean Recovery	N	Range
69	Chrysene-d_{12} (IS)	115	5	96 - 151
70	Terphenyl-d_{14} (BNS)	90	5	69 - 102
71	Benzidine			
72	Pyrene	104	5	90 - 121
73	Benzyl butyl phthalate	103	3	92 - 115
74	Benzo(a) anthracene	105	3	101 - 108
75	3,3'-Dichlorobenzidine			
76	Chrysene	107	3	104 - 109
77	Bis(2-ethylhexyl) phthalate	106	3	95 - 115
78	Di - n - octyl phthalate	113	3	111 - 114
79	Perylene-d12 (IS)	132	4	71 - 210
80	Benzo(b)fluoranthene	104	3	104 - 105
81	Benzo(k)fluoranthene	99	3	95 - 104
82	Benzo(a)pyrene	106	3	105 - 107
83	Indeno(1,2,3-cd)pyrene	107	3	93 - 116
84	Dibenz(a,h)anthracene	112	3	105 - 117
85	Benzo(g,h,i)perylene	108	3	103 - 110

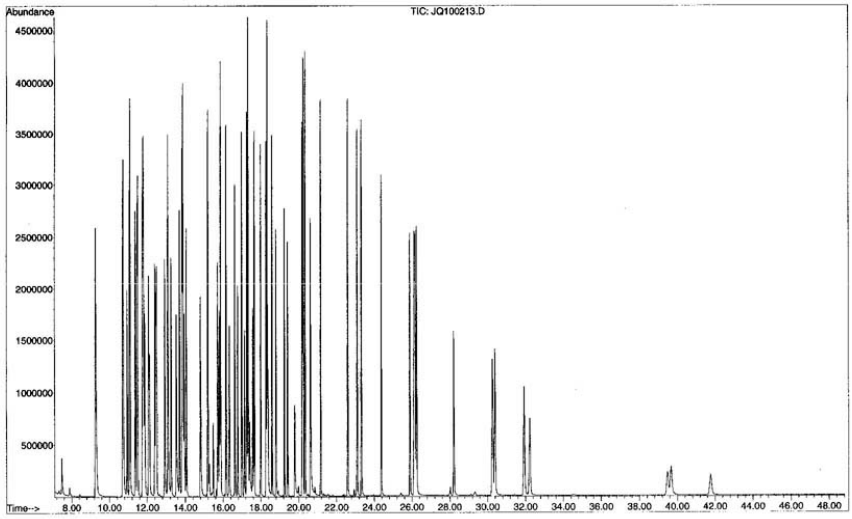
Table 3: Historical tube spike recoveries

		Mean Recovery	N	Range
1	1,4 - dichlorobenzene -d_4 (IS)	104	6	94 - 117
2	2-fluorophenol (AS)	100	7	91 - 107
3	phenol - d_5 (AS)	100	7	94 - 104
4	2-chlorophenol - d_4 (AS)	100	7	97 - 103
5	1,2-dichlorobennzene - d4 (BNS)	100	7	87 - 116
6	N - Nitrosodimethylamine	105	3	100 - 116
7	Aniline			
8	Phenol	95	7	81 - 117
9	Bis(2-chloroethyl) ether	75	3	9 - 116
10	2-Chlorophenol	96	7	82 - 115
11	1,3-Dichlorobenzene	105	3	99 - 115
12	1,4-Dichlorobenzene	100	7	90 - 115
13	1,2-Dichlorobenzene	104	3	99 - 114
14	Benzyl alcohol			
15	2-Methylphenol	106	3	100 - 118
16	Bis(2-chloroisopropyl) ether	105	3	99 - 115
17	4-Methylphenol	105	3	98 - 117
18	Hexachloroethane	110	3	100 - 130
19	N - Nitroso-di - n - propyl amine	97	7	88 - 115
20	Naphthalene - d_8 (IS)	102	6	92 - 116
21	Nitrobenzene - d_5 (BNS)	99	7	81 - 115
22	Nitrobenzene	105	3	100 - 116
23	Isophorone	102	3	99 - 106
24	2-Nitrophenol	107	3	100 - 120
25	2,4-Dimethylphenol	105	3	100 - 114
26	Bis(2-chloroethoxy) methane	105	3	99 - 116
27	Benzoic acid			
28	2,4-Dichlorophenol	106	3	94 - 122
29	1,2,4-Trichlorobenzene	102	7	90 - 116
30	Naphthalene	105	3	99 - 116
31	4-Chloroaniline	104	3	101 - 109
32	Hexachlorobutadiene	106	3	100 - 118
33	4-Chloro-3-methylphenol	99	7	87 - 120
34	2-Methylnaphthalene	105	3	99 - 116

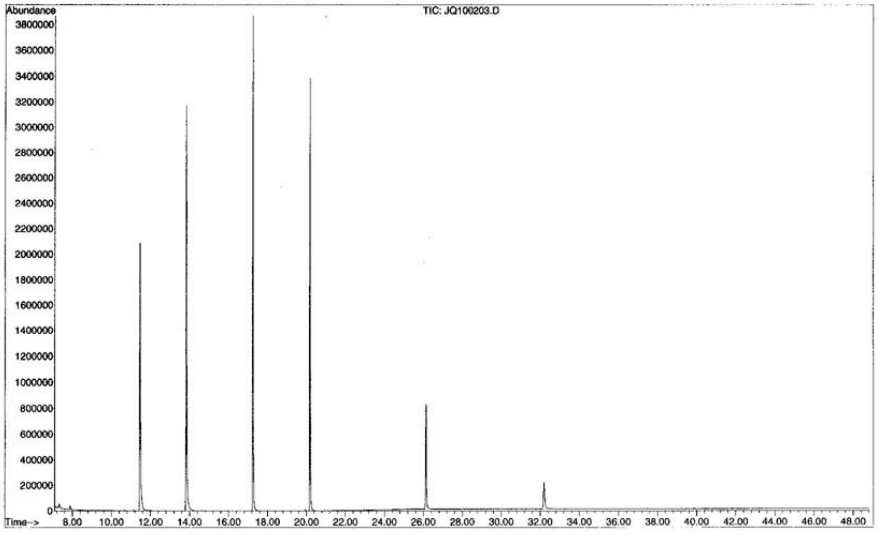
		Mean Recovery	N	Range
35	Acenaphthene-d_{10} (IS)	105	6	93 - 123
36	2-Fluorobiphenyl (BNS)	99	7	82 - 109
37	Hexachlorocyclopentadiene	119	3	111 - 132
38	2,4,6 - Trichlorophenol	107	3	100 - 122
39	2,4,5 - Trichlorophenol	106	3	100 - 117
40	2-Chloronaphthalene	105	3	100 - 113
41	2-Nitroanaline	107	3	101 - 118
42	Dimethylphthalate	105	3	100 - 114
43	Acenaphthylene	103	3	100 - 110
44	2,6-Dinitrotoluene	107	3	100 - 121
45	3-Nitroaniline	107	3	101 - 118
46	Acenaphthene	102	7	90 - 115
47	2,4-Dinitrophenol	109	3	101 - 124
48	4-Nitrophenol	103	7	85 - 131
49	Dibenzofuran	105	3	100 - 116
50	2,4-Dinitrotoluene	96	7	77 - 122
51	Tetrachlorophenol			
52	Diethyl phthalate	105	3	100 - 115
53	Fluorene	105	3	99 - 116
54	4-Chlorophenyl-phenyl ether	105	3	99 - 115
55	4-Nitroaniline	107	3	103 - 114
56	Phenanthrene-d_{10} (IS)	104	6	96 - 122
57	2,4,6-Tribromophenol (AS)	100	7	81 - 109
58	N - Nitrosodiphenylaime			
59	4,6-Dinitro-2-methylphenol	110	3	103 - 123
60	Azobenzene	106	3	100 - 117
61	4-Bromophenyl-phenyl ether	105	3	99 - 115
62	Hexachlorobenzene	104	3	99 - 115
63	Pentachlorophenol	88	7	7 - 122
64	Phenanthrene	106	3	99 - 118
65	Anthracene	105	3	99 - 115
66	Carbazol	106	3	99 - 120
67	Dibutyl phthalate	105	3	98 - 115
68	Fluoranthene	107	3	98 - 122

		Mean Recovery	N	Range
69	Chrysene-d_{12} (IS)	107	6	86 - 141
70	Terphenyl-d_{14} (BNS)	97	7	82 - 113
71	Benzidine			
72	Pyrene	116	7	96 - 149
73	Benzyl butyl phthalate	101	3	99 - 102
74	Benzo(a) anthracene	106	3	101 - 116
75	3,3'-Dichlorobenzidine			
76	Chrysene	107	3	100 - 117
77	Bis(2-ethylhexyl) phthalate	101	3	94 - 106
78	Di - n - octyl phthalate	106	3	105 - 108
79	Perylene-d12 (IS)	102	6	63 - 180
80	Benzo(b)fluoranthene	109	3	105 - 116
81	Benzo(k)fluoranthene	107	3	103 - 113
82	Benzo(a)pyrene	110	3	105 - 118
83	Indeno(1,2,3-cd)pyrene	105	3	93 - 114
84	Dibenz(a,h)anthracene	106	3	97 - 116
85	Benzo(g,h,i)perylene	104	3	99 - 110

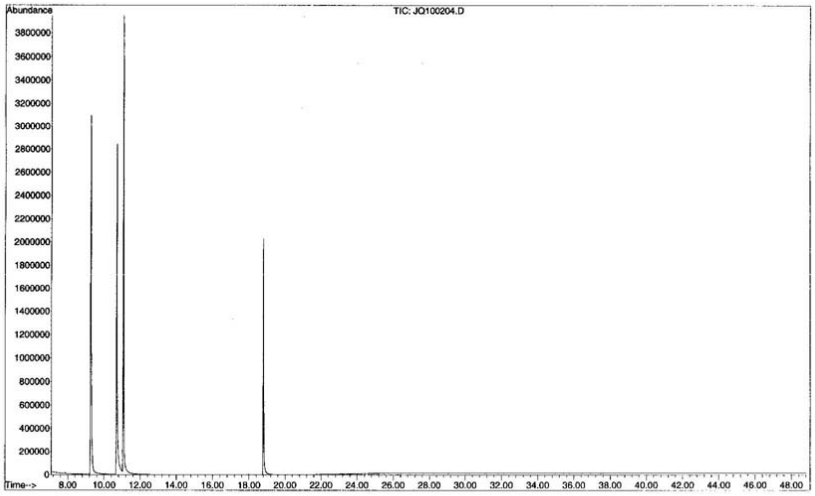
File : D:\DATA\SV021002\JQ100213.D
Operator :
Acquired : 3 Oct 2002 3:18 am using AcqMethod SV021002
Instrument : Instrumen
Sample Name: Std 508-50
Misc Info :
Vial Number: 11



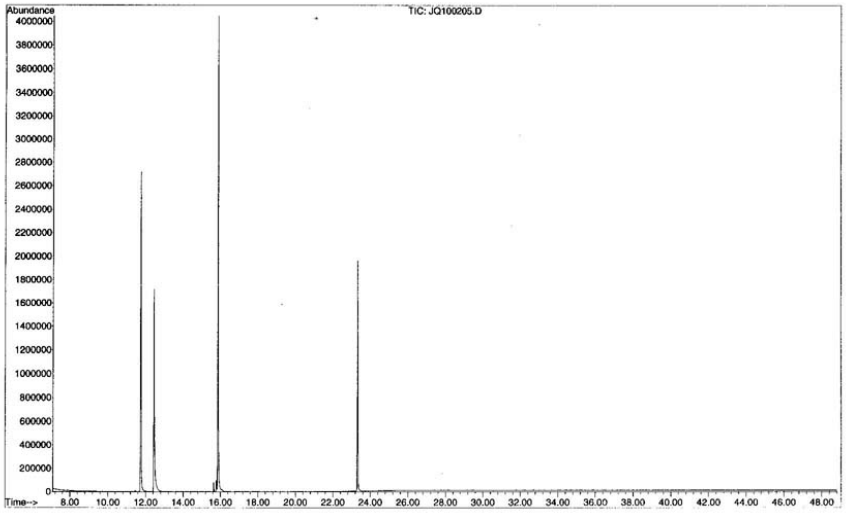
File : D:\DATA\SV021002\JQ100203.D
Operator :
Acquired : 2 Oct 2002 5:02 pm using AcqMethod SV021002
Instrument : Instrumen
Sample Name: SVOC internal standards
Misc Info :
Vial Number: 3



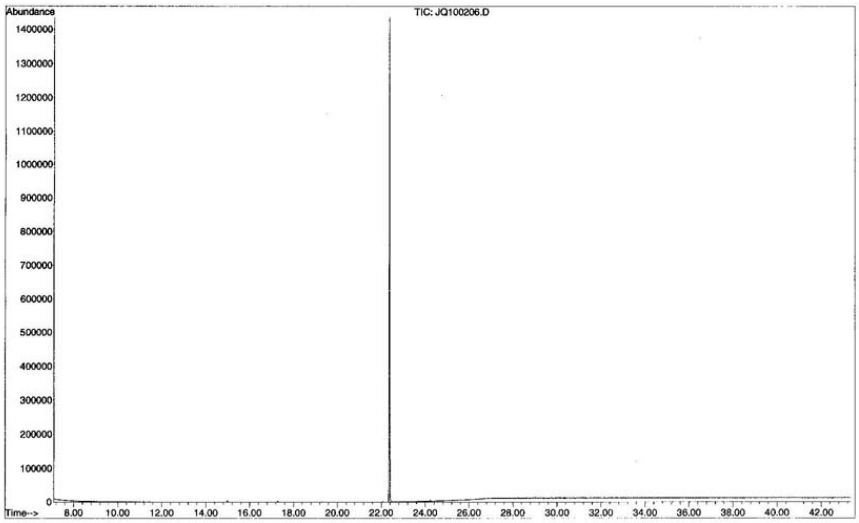
File : D:\DATA\SV021002\JQ100204.D
Operator :
Acquired : 2 Oct 2002 6:04 pm using AcqMethod SV021002
Instrument : Instrumen
Sample Name: SVOC acid surrogates
Misc Info :
Vial Number: 4



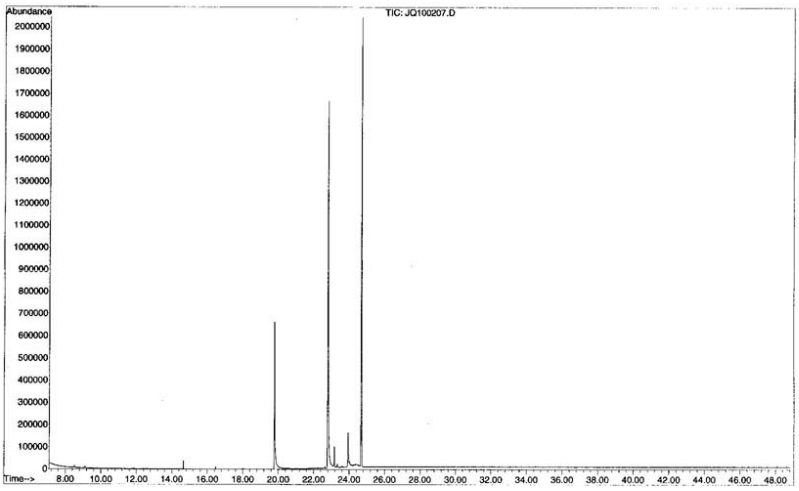
File : D:\DATA\SV021002\JQ100205.D
Operator :
Acquired : 2 Oct 2002 7:06 pm using AcqMethod SV021002
Instrument : Instrumen
Sample Name: SVOC base/neutral surrogates
Misc Info :
Vial Number: 5



File : D:\DATA\SV021002\JQ100206.D
Operator :
Acquired : 2 Oct 2002 8:08 pm using AcqMethod DFTPP
Instrument : Instrumen
Sample Name: DFTPP-1 50 ug/mL
Misc Info :
Vial Number: 6



File : D:\DATA\SV021002\JQ100207.D
Operator :
Acquired : 2 Oct 2002 9:04 pm using AcqMethod SV021002
Instrument : Instrumen
Sample Name: Tune solution
Misc Info :
Vial Number: 7



File : D:\DATA\SV021002\JQ100218.D
Operator :
Acquired : 3 Oct 2002 8:29 am using AcqMethod SV021002
Instrument : Instrumen
Sample Name: LRB 9-17
Misc Info :
Vial Number: 15